



Short communication

Polyethylene-supported polyvinylidene fluoride–cellulose acetate butyrate blended polymer electrolyte for lithium ion battery

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H I G H L I G H T S

- ▶ A novel PE-supported PVDF/CAB polymer membrane was prepared.
- ▶ Excellent electrolyte uptake and ionic conductivity of GPE based on this membrane.
- ▶ Improved cycle, storage and rate performances of lithium ion battery using this GPE.

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The polyethylene (PE)-supported polymer membranes based on the blended polyvinylidene fluoride (PVDF) and cellulose acetate butyrate (CAB) are prepared for gel polymer electrolyte (GPE) of lithium ion battery. The performances of the prepared membranes and the resulting GPEs are investigated by scanning electron microscopy, electrochemical impedance spectroscopy, linear potential sweep, and charge–discharge test. The effect of the ratio of PVDF to CAB on the performance of the prepared membranes is considered. It is found that the GPE based on the blended polymer with PVDF:CAB = 2:1 (in weight) has the largest ionic conductivity ($2.48 \times 10^{-3} \text{ S cm}^{-1}$) and shows good compatibility with anode and cathode of lithium ion battery. The LiCoO₂/graphite battery using this GPE exhibits superior cyclic stability at room temperature, storage performance at elevated temperature, and rate performance.

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1. Introduction

Lithium ion battery has been widely used in portable electronic devices such as cell phone, laptop and camera, because it has many advantages including high energy density and long cycle life [1,2]. However, safety problem exists in current commercial lithium ion battery due to the using of combustible liquid organic electrolyte, especially in large-scale application for electrical vehicles. Gel polymer electrolyte (GPE) that uses polymer matrix to immobilize the liquid electrolyte is a promising solution to the safety problem of lithium ion battery. Therefore, more and more researches are being focused on the development of new GPEs [3,4].

The GPEs based on polyacrylonitrile (PAN) [5,6], poly(vinylidene fluoride) (PVDF) [7–10], poly(ethylene oxide) (PEO), and poly(methyl methacrylate) (PMMA) [11,12], are found to have moderate ion conductivity, $\sim 10^{-3} \text{ S cm}^{-1}$, at room temperature, but their mechanical strength is not strong enough for their applications in lithium ion battery. In order to improve the mechanical strength of GPEs, porous polyethylene (PE) membranes have been used as a dimensional support [9,11]. Blended polymer combines the advantages of different polymers and thus provides GPE with improved performances [12,13]. PVDF is one of the most common components used as the blended polymer because it has high dielectric constant, strongly electron withdrawing functional group (C-F) and good mechanical property. Many polymers, such as poly(ethyl methacrylate) (PEMA) [14], PEO [15], PMMA [16], and copolymer poly(ethylene oxide)–poly(methyl methacrylate) (PEO-PMMA) [17], have been used to blend with PVDF as the matrix of GPEs.

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Cellulose acetate butyrate (CAB) has a special molecular structure (Fig. 1), whose monomer contains acetyl, butyryl and hydroxyl groups. The C=O and C–OH groups can provide polymer with good affinity to electrolyte and good compatibility with electrode. Therefore, good performance of GPE using blended polymer containing CAB can be expected. With the consideration of the advantages of PVDF and CAB, we developed a novel GPE by using a blended polymer of PVDF and CAB. The optimal content of CAB in the blended polymer was determined and the performances of the blended polymer and corresponding GPE for lithium ion battery use were investigated.

2. Experimental

2.1. Preparation

Different ratios (Table 1) of PVDF (MW: 130,000, Arkema Shanghai Co., Ltd.) and CAB (MW: 40,000, Eastman Chemical Company) were dissolved (10 wt.%) in the mixed solvents composed of *N,N*-dimethylformamide (DMF) and dimethyl carbonate (DMC) (DMF:DMC = 9:1, in weight). After vigorous stirring for 5 h, a microporous polyethylene (PE) separator (Celgard 2400, 16 μm thick, Celgard LLC, USA) was immersed in the resulting homogeneous solutions for 15 min, and then taken out and dried in the air atmosphere for 1 h then in the vacuum at 60 °C for 24 h. The PE-supported PVDF-CAB blended polymer membranes with a final thickness of about 35 μm , measured with a digital micrometer caliper (Cal PRO IP67, SYLVAC, Swiss), were obtained. To prepare GPEs, the PE-supported blended polymer membranes were soaked with a liquid electrolyte for 40 min. The liquid electrolyte is 1 mol L⁻¹ LiPF₆ in the mixed solvent of ethylene carbonate (EC) and ethyl methyl carbonate (EMC) (EC:EMC = 2:3 in weight, battery grade, Guangzhou Tinci Material Technology Co., Ltd).

2.2. Characterization

The viscosity of the blended polymer in DMF/DMC was measured by using a viscosimeter (RVT, Brookfield, USA) at room temperature. The surface morphology of the prepared membranes and the average pore size of the membranes were measured by scanning electron microscopy (SEM, JSM-6380LV, JEOL, Japan). The electrolyte uptake (A%) of the prepared membranes was obtained by [11]:

$$A\% = (W_2 - W_1)/W_1 \times 100\% \quad (1)$$

where W_1 and W_2 stand for the mass of the prepared membrane before and after soaking the liquid electrolyte, respectively.

To measure the porosity, the prepared polymer membranes with different ratios of PVDF to CAB were immersed into *n*-butanol for 2 h. The porosity (P) was obtained by Eq. (2).

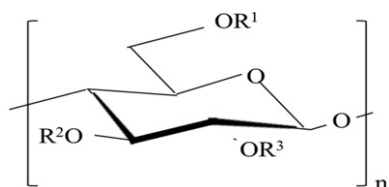


Fig. 1. Molecular structure of CAB, R¹, R² and R³ represent acetyl, butyryl and H, respectively.

Table 1

Physical properties of the blended polymer with different ratios of PVDF to CAB at room temperature.

PVDF/CAB (in weight)	1:1	2:1	3:1	4:1
Viscosity (mPa s)	905	1083	1140	1298
Electrolyte uptake (%)	180	152	148	128
Pore diameter (μm)	2.35	1.91	1.72	1.13
Porosity (%)	46.3	40.7	37.0	30.5
Conductivity (mS cm ⁻¹)	2.05	2.48	1.98	0.72

$$P = \frac{m_a/\rho_a}{(m_a/\rho_a) + (m_p/\rho_p)} \quad (2)$$

where ρ_a and ρ_p are the density of *n*-butanol and the dry membrane, respectively; m_a and m_p are the mass of the *n*-butanol incorporated membrane and the dry membrane, respectively.

The ionic conductivity of the GPE was measured with blocking-type cell, in which the GPE was sandwiched between two stainless steel (SS) electrodes. Impedance data were obtained on Instrumental Electrochemical Workstation (CHI660, Chenhua, Shanghai) using frequencies ranging from 1 Hz to 100 kHz and with potential amplitude of 5 mV. The ionic conductivity of the GPE was obtained by:

$$\sigma = L/(A \cdot R) \quad (3)$$

where L is the thickness of the GPE, A is the contact area between the GPE and SS, and R is the bulk resistance of the GPE.

The compatibility of the GPE with cathode and anode of lithium ion battery was understood by measuring the oxidation decomposition potential of the GPE and the interfacial impedance between the GPE and lithium, respectively. The oxidation decomposition potential of the GPE was determined on SS electrode by linear sweep voltammetry with lithium foil as the counter electrode and reference electrode. The interfacial impedance was determined on lithium foil electrode by electrochemical impedance spectroscopy.

To determine the battery performance, rectangular battery was assembled with LiCoO₂ (Hunan Reshine New Material Co., Ltd) as cathode, artificial graphite (BTR Battery Materials Co., Ltd) as anode. The LiCoO₂ electrode was prepared by mixing 93.8 wt.% LiCoO₂, 3.0 wt.% carbon and 3.2 wt.% binder PVDF and coating the slurry onto an Al foil. The graphite electrode was prepared by mixing 95.0 wt.% graphite, 1.0 wt.% acetylene black, 1.5 wt.% carboxy methyl cellulose, and 2.5 wt.% styrene butadiene rubber and coating the slurry onto a copper foil. Charge–discharge test of the battery was conducted on BS 9300R (Qingtian, Guangzhou), between 2.75 V and 4.2 V at 1C rate.

3. Results and discussion

3.1. Optimal content of CAB in blended polymer

To obtain the optimal content of CAB in the blended polymer, the viscosity of the blended polymer, the electrolyte uptake and porosity of the membrane and the ionic conductivity of the GPEs were determined. Table 1 presents the obtained results.

It can be seen from Table 1 that the viscosity of the gel polymer increases with increasing content of PVDF. This should be ascribed to larger molecule weight of PVDF and the stronger interaction of PVDF with its strong electron withdrawing group C–F [7]. The viscosity of the gel polymer is important for the formation of the polymer membrane with good performance.

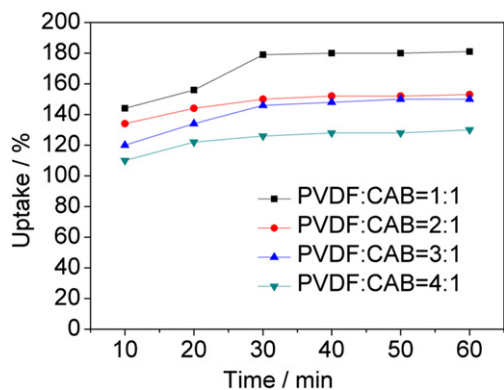


Fig. 2. Electrolyte uptake of the PE-supported polymer membranes with different ratios of PVDF to CAB (in weight).

Fig. 2 displays the variation of electrolyte uptake of the membranes with soaking time. The electrolyte uptake increases quickly at the first stage of soaking, keeps almost unchanged after 30 min and reaches maximum at 40 min. The maximal electrolyte uptake of the membranes is included in Table 1. It can be seen from Table 1 that the electrolyte uptake and the porosity of the membranes increase with increasing CAB content. The difference in electrolyte uptake and the porosity should be related to the different pore structure of the membranes.

Fig. 3 presents the SEM images of various membranes. It can be seen from Fig. 3 that the pore size of the PE support is small (Fig. 3a), while larger pores are formed on the blended polymer membranes (Fig. 3b–e). The pores are interconnected in the blended polymer, which is important for the ionic transportation. It can be noted that the pore distribution and structure size are affected by CAB content. The porosity and the pore diameter of the membrane increase with increasing the CAB content. This should be ascribed to the decreased viscosity of the blended polymer when increasing the content of CAB in polymer. During the preparation of polymer membrane, the lower viscosity of the polymer slurry favors the improving of porosity and the formation of larger pore [18–21]. This result explains why the membrane with the blended polymer of PVDF:CAB = 1:1 has the maximum electrolyte uptake. The average pore size of the membranes is included in Table 1. Obviously, CAB improves the pore structure of PVDF-based membrane. The pore size of a pure PVDF membrane is usually less than 0.5 μm [22]. It can be seen from Table 1 that there is a proportional relation of electrolyte uptake with the porosity and the pore size.

The ionic conductivity of the polymer membrane plays an important role in rate performance of a lithium ion battery [23,24]. Table 1 presents the ionic conductivity of the GPEs with different CAB contents at room temperature, obtained by impedance measurement. Fig. 4 presents the Nyquist plots of SS/PE-supported GPEs /SS cell with different ratios of PVDF to CAB at various temperature. As can be seen in Fig. 4(a), the real part and imaginary part of the impedance are in a linear relationship, reflecting the

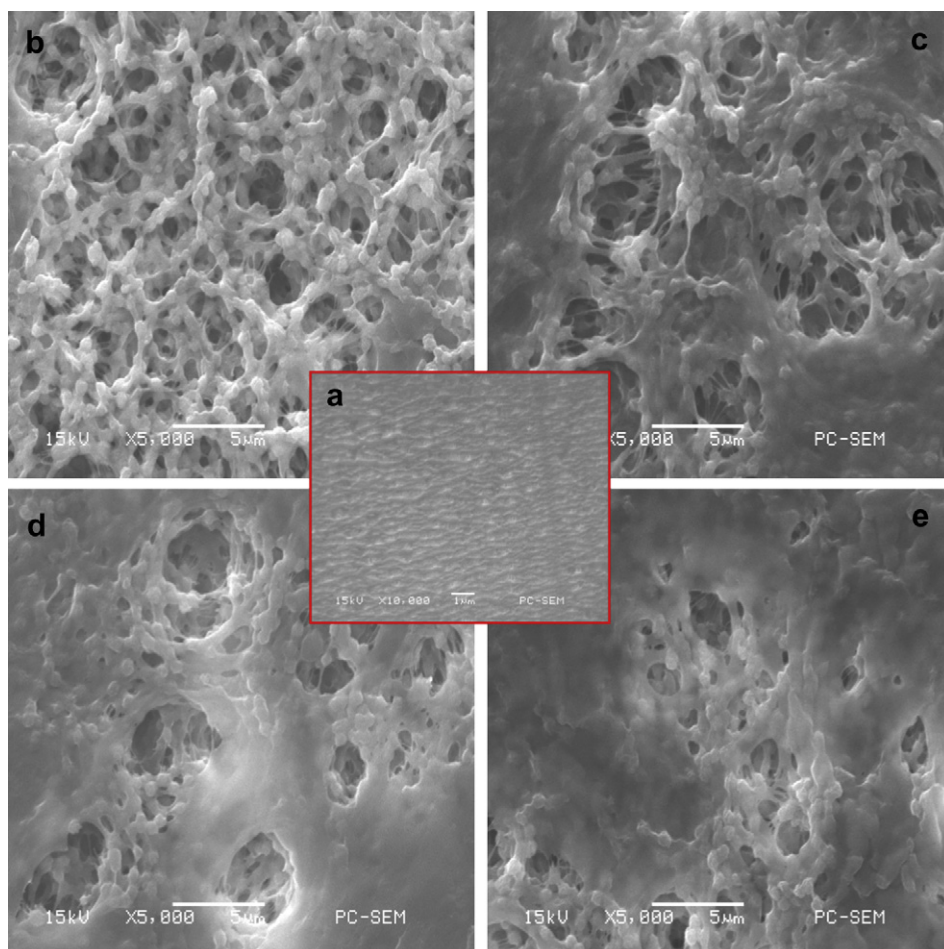


Fig. 3. SEM images of the PE membrane (a) and the PE-supported polymer membranes with different ratios of PVDF to CAB: (b) 1:1, (c) 2:1, (d) 3:1 and (e) 4:1.

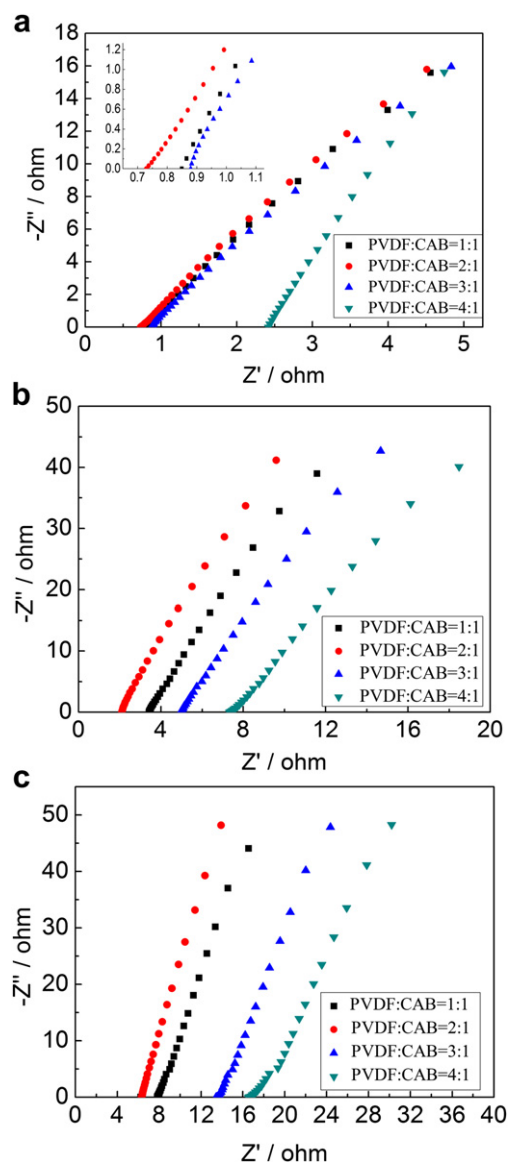


Fig. 4. Nyquist plots of SS/PE-supported GPEs /SS cell with different ratios of PVDF to CAB at various temperature (a) room temperature, (b) 0 °C, and (c) -20 °C.

lithium ion transportation in the GPE. The intersection between the linear line and the real axis is the resistance for the ionic conductivity calculation (Eq. 3). It can be seen from Table 1 that the ionic conductivities of the GPEs with PVDF:CAB = 1:1, 2:1 and 3:1 are about 2 mS cm^{-1} . These values are higher than other reported PVDF-blended systems. For example, the conductivity of GPEs with PVDF/poly(dimethylsiloxane) (PDMS) blended polymer was 1.17 mS cm^{-1} [25], and that with PVDF/PMMA blended polymer was 1.21 mS cm^{-1} [26]. It can be noted that the GPE with PVDF:CAB = 2:1 has larger ionic conductivity than that with PVDF:CAB = 1:1, although the later has larger electrolyte uptake than the former. This indicates that the larger electrolyte uptake does not mean larger ionic conductivity. The larger ionic conductivity of the GPE with PVDF:CAB = 2:1 might be related to the better pore structure inside the membrane. The larger content of CAB (PVDF:CAB = 1:1) results in the lower viscosity of blended polymer slurry for membrane preparation and might not favor the formation of interconnected pore inside the membrane, which plays very important role in ionic conduction of the resulting GPE.

Similar results were obtained under low temperature. Fig. 4(b) and (c) presents the Nyquist plots of the GPEs with different ratios of PVDF to CAB at 0 °C and -20 °C, respectively. As the temperature decreases, the ionic conductivity of the GPEs tends to decline. Nevertheless, the GPE with PVDF:CAB = 2:1 has the lowest impedance among all the GPEs, and the ionic conductivity of which is 0.83 mS cm^{-1} at 0 °C and 0.27 mS cm^{-1} at -20 °C, respectively (calculated by Eq. (3)). Therefore, the GPE with PVDF:CAB = 2:1 is good for use in the lithium ion battery at low temperature.

3.2. Compatibility of GPE with anode and cathode of lithium ion battery

Fig. 5 shows the linear sweep voltammograms of SS electrodes in Li/PE-liquid electrolyte/SS and Li/PE-supported GPE (PVDF:CAB = 2:1)/SS cells. It can be seen from Fig. 5 that liquid electrolyte decomposes at 4.4 V, while the GPE is stable until 4.7 V. This indicates that the GPE has the better stability on or better compatibility with cathode of lithium ion battery. The free solvent molecules in liquid electrolyte tend to be decomposed on cathode of lithium ion battery [27,28], but their stability can be improved by gelation with polymer [29]. This may be ascribed to the interaction between the solvent molecules and the blended polymer, which results from the combination between oxygen atoms in polymer and hydrogen atoms in solvent molecules. The oxidative decomposition of solvents is the main cause for the safety problem of lithium ion battery. This is why GPEs are developed for lithium ion battery use.

Fig. 6 shows the Nyquist plots of Li/PE-liquid electrolyte/Li and Li/PE-supported GPE (PVDF:CAB = 2:1)/Li cells for understanding the compatibility of the GPE with the anode of lithium ion battery. The diameter of the semicircle in the plots reflects the interfacial resistance between lithium and electrolyte. It can be seen from Fig. 6 that the interfacial resistance increases quickly in the first 20 days, while slowly after 20 days. Compared to the cell using PE-liquid electrolyte (Fig. 6a), the cell using PE-supported GPE (PVDF:CAB = 2:1) (Fig. 6b) has lower interfacial resistance, suggesting that less ability of the GPE to react with lithium, or better compatibility with anode of lithium ion battery than the liquid electrolyte. The compatibility improvement results also from the enhanced stability of the solvents due to the gelation.

3.3. Battery performance

Fig. 7 shows the cyclic performances of LiCoO₂/PE-liquid electrolyte/graphite and LiCoO₂/PE-supported GPE /graphite batteries at room temperature. It can be seen from Fig. 7 that the capacity

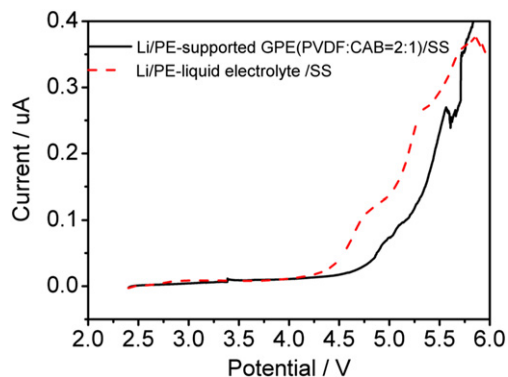


Fig. 5. Linear sweep voltammograms of SS electrodes in Li/PE-liquid electrolyte/SS and Li/PE-supported GPE (PVDF:CAB = 2:1)/SS cells. The scan rate is 1 mV s^{-1} .

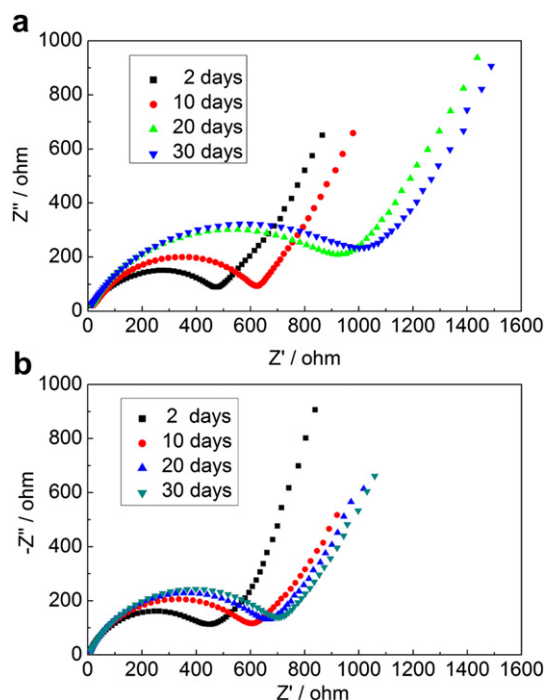


Fig. 6. Nyquist plots of (a) Li/PE-liquid electrolyte/Li and (b) Li/PE-supported GPE (PVDF:CAB = 2:1)/Li cells at open circuit potential.

retention is related to the ratios of PVDF to CAB. The battery using PE-supported GPE (PVDF:CAB = 2:1) shows the highest capacity retention (93.1% after 300 cycles), while the capacity retention of the batteries decreases with increasing or reducing the CAB content. Many factors affect the cyclic performance of lithium ion battery, including the stability of electrode materials and electrolyte and the ionic conductivity of electrolyte. Considering the lower capacity retention (80.2%) of the battery using PE-liquid electrolyte, it can be known that the better cyclic performance of the battery using the GPE with PVDF:CAB = 2:1 results from the improved stability of the electrolyte. On the other hand, better performance of the battery using the GPE with PVDF:CAB = 2:1 than those of the batteries using other ratios of PVDF to CAB results mainly from the larger ionic conductivity of the GPE. Compared with the PE-supported pure PVDF-based GPE (only 86% capacity retention after 100 cycles for the battery using this GPE) [30], it is obvious that the application of CAB improves significantly the cyclic performance of the battery using PVDF-based GPE.

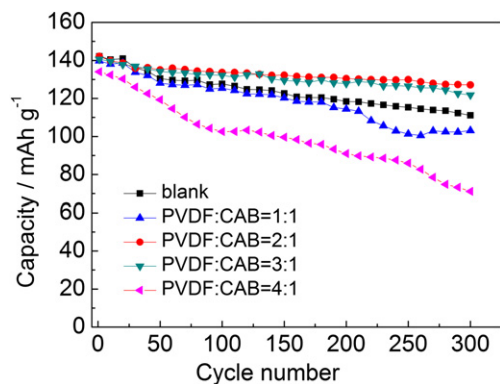


Fig. 7. Cycle performances of LiCoO₂/PE-liquid electrolyte/graphite and LiCoO₂/PE-supported GPEs with different ratios of PVDF to CAB/graphite batteries, charged and discharged with 1C rate at room temperature.

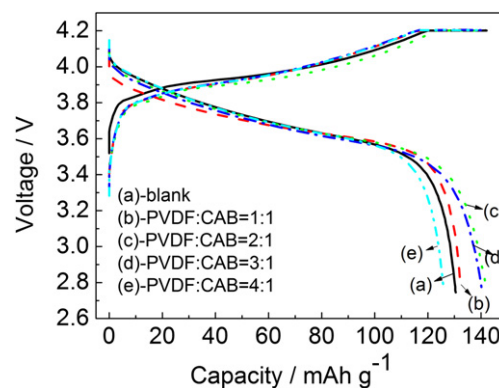


Fig. 8. Charge and discharge curves of the batteries using PE-liquid electrolyte and PE-supported GPEs with different ratios of PVDF to CAB at 1C rate. Before the charge–discharge test, the batteries were fully charged and then stored at high temperature (75 °C) for 48 h.

Fig. 8 displays the charge and discharge curves of the batteries using PE-liquid electrolyte and PE-supported GPEs after full charged and then stored at high temperature (75 °C) for 48 h. It can be seen from Fig. 8 that the battery using the GPE with PVDF:CAB = 2:1 exhibits the best high temperature storage performance and the effect of CAB is similar to that on cyclic performance, indicating that the PE-supported GPE (PVDF:CAB = 2:1) improves the storage performance of lithium ion battery.

Similar result was also observed for the effect of CAB on the rate performance of the battery. Fig. 9 presents the rate performances of the batteries using PE-liquid electrolyte and PE-supported GPEs. The battery using PE-supported GPE (PVDF:CAB = 2:1) delivers the highest discharge capacity, especially at high rate. At 5C, the battery using PE-supported GPE (PVDF:CAB = 2:1) keeps 87.8% of its 0.2C discharge capacity. This value is far larger than those that have been reported. For example, the 2C capacity of the battery using PE-supported GPE with pure PVDF keeps only 55.6% of its 0.2C capacity [22] and the value for the battery using PE-supported GPE with PVDF-HFP is only 70.5% [31].

The improved battery performance by using the GPE should be ascribed to the appropriate ionic conductivity of the GPE and the superior compatibility of the GPEs with the anode and cathode of lithium ion battery compared to the liquid electrolyte. The gelation of the liquid electrolyte reduces the decomposition of solvents or salt on anode and cathode of lithium ion battery, inhibits the growth of the passive films on anode and cathode, and thus improves the battery performances.

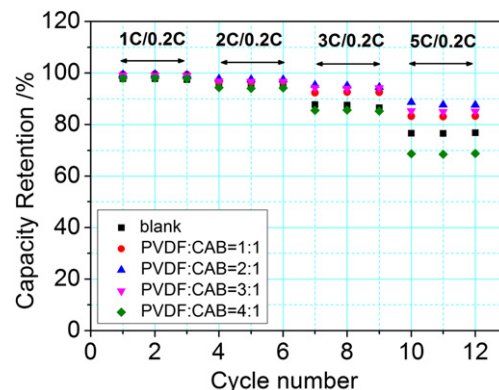


Fig. 9. Rate performances of the batteries using PE-liquid electrolyte and PE-supported GPEs with different ratios of PVDF to CAB.

4. Conclusions

A novel PE-supported PVDF-CAB blended polymer membrane has been prepared. With increasing the CAB content in blended polymer, the viscosity of gel polymer decreases and electrolyte uptake of the membrane increases. The GPE based on the supported membrane with PVDF:CAB = 2:1 has the largest ionic conductivity. LiCoO₂/graphite battery using this GPE exhibits better cycle, storage and rate performances than the battery using liquid electrolyte. The improved battery performances are attributed to the better compatibility of the GPE with anode and cathode of lithium ion battery.

Acknowledgments

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